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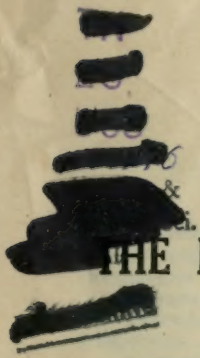

THE DETECTION AND ESTIMATION OF PLATINUM IN ORES

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ENGINEERING

BY

C. W. DAVIS

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THE DETECTION AND ESTIMATION OF PLATINUM IN ORES.

BY

C. W. DAVIS.

INTRODUCTION.

The present high price of platinum has stimulated the prospector to a greater activity in the search for new occurrences of this metal. Inexperienced assayers have been called upon to examine ore samples for platinum and have submitted results of no value, either through errors in analysis or through an attempt to deceive.

Some of the companies that have been organized for the purpose of recovering platinum from ores and sands declare that certain forms of platinum in an ore can not be removed or determined by the usual methods of fire assay or "wet" treatment, but can be recovered by special methods. The results given in this paper corroborate the opinion of reliable chemists who have had experience in the assay of material containing platinum (see pp. 10–11) that such reports are unconfirmed and should be regarded with suspicion.

During the World War the examination of platinum ores was assigned to the Golden station of the United States Bureau of Mines, and since then the station has received numerous inquiries concerning the commercial assay of ores for platinum. Although a mass of information concerning analyses of platinum has been published, it is by no means easy to select a satisfactory method for the commercial assay of ores containing small amounts of platinum.

This paper has been prepared to furnish a ready reference to those assayers who have had difficulty in detecting or determining platinum; it summarizes methods for the detection of the metal and gives a selected method for the commercial estimation of platinum in ores. The separation and determination of the various platinum metals—ruthenium, rhodium, palladium, osmium, iridium, and platinum—is at best difficult, requiring the services of a specialist, and can not be done with the small quantities of metal found in the analysis of an ore.

ACKNOWLEDGMENTS.

The author wishes to express his appreciation for the helpful suggestions of S. C. Lind and L. H. Duschak, of the United States Bureau of Mines, and to acknowledge gratefully the hearty cooperation of Dr. James Lewis Howe, Mr. Frederic P. Dewey, Mr. L. W. Dunham, and Mr. Abbott A. Hanks.

OCCURRENCE OF PLATINUM.

MINERALS.

Platinum occurs native as an alloy containing different quantities of the platinum metals, iron, and copper in small steel-gray, flattened grains or pellets of specific gravity 16 to 19, or more rarely as nuggets in alluvial deposits resulting from the disintegration of peridotite, gabbro, and serpentine. Small quantities of the platinum metals exist in sulphide ores of copper and nickel. The precious metals accompany the copper in the smelting process and are eventually recovered from the sludge that is formed when the latter is refined electrolytically.

The only compound of platinum found in nature is sperrylite, the diarsenide, $PtAs_2$.

DISTRIBUTION.[1]

The principal foreign deposits of platinum include those of the Ural Mountains, Russia; the United States of Colombia, South America; the Ronda Mountains, Spain; New South Wales, Australia; Borneo; Griqualand, South Africa; Tasmania; Sumatra; Brazil; New Zealand; France; Norway; and Canada. Platinum also occurs in Alaska, and in Trinity, Siskiyou, Calaveras, Yuba, Nevada, Butte, Humboldt, Merced, Del Norte, and San Bernardino Counties, Calif.; in Coos and Curry Counties, Oreg.; in Clark County, Nev.; in Laramie County, Wyo.; in Boulder and Saguache Counties, Colo.; in Lancaster and Berks Counties, Pa.; in Macon County, N. C.; and in the Grand Canyon of the Colorado River.

Although small amounts of the platinum metals have been found thus widely distributed, about 99 per cent of the platinum recovered has come from placer workings, chiefly those of the Ural Mountains and the United States of Colombia.

PROPERTIES.

Metallic platinum is white, malleable, and rather soft, having a specific gravity of 21.45. It melts at 1,755° C. and can be welded at white heat. Single acids do not attack pure platinum in the cold, but hot sulphuric acid dissolves the finely-divided metal to some extent. Aqua regia readily dissolves it, and fusions with alkaline hydroxide, peroxide, nitrate, or cyanide attack it appreciably. Nitric acid dissolves platinum alloyed with much silver, copper, or lead. Platinum readily alloys with gold and the platinum metals as well as with silver, lead, bismuth, antimony, and arsenic.

DETECTION.

For all practical purposes, the detection and the estimation of platinum in ores overlap, owing to the fact that the platinum is often present in too small quantities for any "rough" method to give satisfactory results. Then, too, after the estimation has been

[1] Uglow, W. L., Geology of platinum deposits: Eng. and Min. Jour., vol. 108, 1919, p. 352.

made, it is well to examine the resulting material qualitatively. The usual procedure is to run a quantitative analysis (as given in a following section of this paper) to the point where the platinum is separated from the other elements, and then to apply a qualitative test. If the platinum is in the metallic state it is dissolved in aqua regia, evaporated just to dryness on a water bath (heating in air will decompose all platinum salts and give the metal) taken up with HCl, and again evaporated to dryness to remove the last traces of HNO_3. The residue of chloroplatinic acid is then dissolved in water with the addition of a few drops of HCl, and a reagent added which will give a characteristic reaction with platinum.

If considerable quantities of the platinum metals are present, the following general scheme, taken from Schoeller and Powell,[2] will be useful in detecting them:

Platinum metals may be detected by the "glow reaction" of Curtman and Rothberg as used by Logan.[2a] The test depends on the catalytic effect of finely divided platinum metal in the oxidation of illuminating gas. The material to be examined is dissolved in aqua regia and the test applied without any previous separation of elements. The test is more sensitive if the solution is not too acid. A very thin piece of asbestos paper, held by one end with tongs, is alternately dipped into the solution to be tested and heated until about 0.2 cubic centimeter has been absorbed. The asbestos is then heated to redness, cooled until the redness is no longer visible, and then thrust at once, while still hot, into a stream of mixed gas and air from a Bunsen burner which is regulated to deliver the mixture at a rather low pressure. If platinum is present the asbestos glows for some time. When destroyed the property of glowing can be restored by heating to redness again. The test is made more intense by using warmed gas and is said to be sensitive to 0.002 milligram of platinum, 0.005 milligram of iridium, 0.0009 milligram of rhodium, and 0.0005 milligram of palladium, but does not reveal the presence of osmium or ruthenium.

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[2] Schoeller, W. R., and Powell, A. R., The analysis of minerals and ores of the rarer elements. 1919, p. 227.

[2a] Logan, C. A., Platinum and allied metals in California: Cal. State Min. Bur., Bulletin 85, 1919, p. 102.

TABLE 1.—*Detection of platinum by Schoeller and Powell method.*

Fuse with litharge and fluxes (as in assay for gold and silver), cool and break crucible, clean button, dissolve in HNO_3 (1:10), and filter.

<table>
<tr><td colspan="3">F (1): Ppt. with H_2SO_4, filter, reject ppt., treat filtrate with a little HCl, filter.</td><td colspan="3">R (1): Warm with dilute aqua regia, filter.</td></tr>
<tr><td colspan="2">P (2): AgCl.</td><td>F (2): Evap. to fumes, filter off, and reject $PbSO_4$, treat filt. with AgI, or a drop of KI solution; Pd will give a black ppt.</td><td>R (3): Fuse with $KOH+KNO_3$ in Ag crucible, leach melt, decant, clear solution into distilling flask, wash residue.[a]</td><td>F (3): Evap. with HCl, take up with little water, sat. with NH_4Cl, let set, filter.</td></tr>
<tr><td colspan="2">S (6): Acidify HCl, distill with a little HNO_3 into KOH solution.</td><td>R (6): Extr. with dil. HNO_3, heat residue with NaCl in Cl current. Dissolve in little water, sat. NH_4Cl; reddish-black ppt. shows Ir.</td><td>P (4): Yellow ppt. shows Pt.</td><td>F (4): Boil with oxalic acid, filter.</td></tr>
<tr><td>D (7): Add C_2H_5OH and heat; a purple color indicates Os.</td><td>S (7): Make alkaline with NH_4OH, add $Na_2S_2O_3$, warm; a violet color indicates Ru.</td><td></td><td>F (5): Add H_2SO_4, reject any $PbSO_4$. Ppt. Rh. with Zn, heat ppt. with NaCl in Cl current; add drop of water to melt; rose-red color shows Rh.</td><td>P (5): Au.</td></tr>
</table>

[a] Note to R(3): The fusion may also be carried out with NaOH and Na_2O_2 in a nickel crucible. In this case the residue R(6) from the decantation is dissolved in hot dilute HCl, the solution treated with $NaNO_3$ and Na_2CO_3, the filtrate from the nickel precipitate evaporated with HCl and tested with NH_4Cl for iridium.

REACTIONS.

The following characteristic reactions have been used for the detection of platinum when in solution as chloroplatinic acid.

Ammonium and potassium chlorides produce in concentrated solutions yellow precipitates—$(NH_4)_2PtCl_6$ and K_2PtCl_6. Both salts are with difficulty soluble in water, but practically insoluble in 75 per cent ethyl alcohol and in concentrated solutions of potassium and ammonium chlorides (difference from gold).

Alkali iodides give a brownish-red coloration due to the formation of the less ionized PtI_6 radical. This test, using potassium iodide, is the one generally employed for the detection of platinum. Field [3] found that 0.000.0005 gram of platinum could be detected by this test.

Tellurium gives a red color with potassium iodide but differs from platinum in that the color disappears on boiling.

Copper solutions give red colors with potassium iodide which might easily be mistaken for platinum reactions.

Hydrogen sulphide precipitates very slowly in the cold, but quickly on warming, dark-brown platinum disulphide (PtS_2); the precipitation is not complete, however. Platinum sulphide is insoluble in pure, single mineral acids, but readily soluble in aqua regia. It is soluble with difficulty in alkali sulphides, but more readily soluble in alkali polysulphides, forming a thio salt, which is decomposable by acids, with the precipitation of platinum sulphide.

Ferrous salts do not reduce chloroplatinic acid in the presence of acids (difference from gold), but cause the precipitation of all the platinum, on warming, in a solution which has been neutralized with sodium carbonate.

Oxalic acid does not precipitate platinum (difference from gold).

Formic acid precipitates from neutral, boiling solutions all the platinum in the form of a black powder. (An acid solution is best neutralized with sodium carbonate before this test.)

Stannous chloride reduces chloroplatinic acid to chloroplatinous acid (H_2PtCl_4) only, not to metal. A brown or red color (yellow when in small amounts) indicates the platinum. The author has found it possible to detect 0.000,002,5 gram of platinum in one cubic centimeter by this test. (See colorimetric method, p. 15.)

Glycerol and sodium hydroxide when warmed throw out black, pulverulent metal.

Carbon monoxide, when passed into a solution of chloroplatinic acid containing some sodium acetate, colors the solution red, owing to the formation of colloidal platinum. After this stands some time all the platinum is precipitated as a black powder, leaving the supernatant liquid colorless.

Zinc, cadmium, magnesium, or aluminum reduces chloroplatinic acid to the metal. The precipitated metal is in such a finely divided or colloidal condition that it tends to run through the filter, especially when washed with pure water. By washing with salt solution a clear filtrate can be obtained.

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[3] Rüdisüle, A., Nachweis, Bestimmung und Trennung der Chemischen Elemente: Bd. 2, 1913, p. 68.

Hydrazine hydrochloride, $N_2H_4.2HCl$, has been used by Rupp.[4] It readily reduces chloroplatinic acid in ammoniacal solutions; the platinum is deposited partly as a mirror upon the sides of the vessel containing the solution.

Formaldehyde in alkaline solutions precipitates the platinum as extremely fine platinum-black. The precipitate may be washed with alkali salt solutions, but with pure water a black colloidal solution of platinum is formed.

Benzidine in a solution of 20 per cent acetic acid, when added to a dilute platinum solution, gives a blue flocculent precipitate. This test is sensitive to 0.000,012,5 gram of platinum per cubic centimeter of solution, but is not applicable in the presence of other metals.[5]

Bannister and Patchin[6] state that under a low-power microscope ($\frac{3}{4}$ or 1 inch objective) the pure silver bead obtained from a fire assay shows crystals arraigned in sets of lines, while a bead containing platinum has a corrugated appearance. A bead with less than 0.3 per cent of platinum appears the same as a silver bead. A bead with 1.6 per cent or more of platinum shows a corrugated surface to the unaided eye.

ESTIMATION OF PLATINUM.

Methods for the determination of platinum in ores may be separated into two classes. The first includes those known as "wet" methods of analysis. These are used on concentrates and material containing considerable quantities of the platinum metals and are not applicable to the estimation of platinum in low-grade ores. The second class—fire-assay methods—are the only ones available at the present time for the determination of small amounts of platinum in vein material. Although claims have been made that ore has been found in which the platinum occurs in such a form as to escape detection by either the "wet" treatment or the fire assay, the author doubts the existence of such material.

In connection with this matter, Ledoux & Co., of New York, say: "So far as our experience goes, platinum metals can always be detected by fire assay." James M. Hill[8] gives his opinion of platinum ores in which "the platinum could not be detected by the ordinary methods of assay," in the following sentence: "From all available information such reports should be regarded with suspicion, for it is confidently believed that any platinum ore of commercial grade will yield to standard methods of analysis if made by competent and reliable analysts." Frederic P. Dewey, chief assayer, Bureau of the Mint, Washington, D. C.,[9] says: "Methods which would unlock platinum not usually disclosed by fire assay I class in the same category with gold making. * * * I have also had considerable experience with these platinum fakes." L. W. Dunham, of Irvington, N. J., who has had much experience in the analysis of

[4] Rüdisüle, work cited.
[5] Malatesta, G., and Nola, E., Detection of gold and platinum by benzidine: Jour. Chem. Soc., vol. 104, ii, 1913, p. 883.
[6] Bannister, C. O., and Patchin, G., Detection of platinum metals in cupellation beads: Jour. Chem. Met. Min. Soc., South Africa, vol. 14, 1914, p. 478.
[7] Private communication, 1920.
[8] Hill, James M., Mineral resources of the United States: 1916, p. 7.
[9] Private communication, 1920.

platinum ores, says:[10] "As to the occurrence of platinum in any ore in such a state as to defy removal by fire assay, I should say there is none. Fire assay is a last resort." Dr. James Lewis Howe, of Washington and Lee University, Lexington, Va., in charge of platinum for the War Minerals Board, writes:[11] "Personally, I have no faith in the existence of platinum in nature (or elsewhere) that can not be detected in the course of a careful assay (fire). Appreciable loss by volatilization in assaying is unthinkable," and, speaking of colloidal platinum in nature, says: "However, granting the possibility of all this, it would seem improbable that even in such a colloidal state the metal should, at assay temperature, altogether or even largely escape solution in the lead."

In tests to determine whether colloidal platinum would be recovered by the fire assay, weighed amounts of platinum were added to regular crucible charges consisting of silica, litharge, sodium carbonate, borax glass, charcoal (to give a 30-gram button), and 0.05 gram of silver chloride. The platinum was introduced in four ways: The first was to wrap a small piece of platinum in lead foil and to add it to the charge; the second, to mix finely divided platinum (obtained by boiling a solution with formic acid to precipitate the platinum) with the charge; the third, to add sodium silicate to a solution of chloroplatinic acid, with hydrogen sulphide added in the cold to form a colloid, and with the addition of enough ammonia to form a gelatinous precipitate, the mixture being dried and mixed thoroughly with the flux; the fourth, to repeat the third method, omitting the addition of hydrogen sulphide.

The fusions were conducted in a gasoline furnace at a temperature of about 1,200° C., and in all tests the platinum was recovered without difficulty, as shown below:

Recovery of platinum by fire assay.

Method of preparation.	Milligram of platinum added.	Milligram of platinum recovered.
First	0.52	0.51
Second	.32	.33
Third	.50	.49
Fourth	.35	.35

The results of these experiments, coupled with similar results obtained by such reputable assayers as Abbott A. Hanks, of San Francisco, and W. L. Piers, of Denver (who were able to recover known weights of finely divided platinum which had been added to regular crucible charges and analyzed by the fire-assay method without an abnormally high fusion temperature), not only show the possibility of the recovery of colloidal platinum by the fire assay, but also indicate that excessive temperatures (those not attainable in the gasoline furnace) are not essential to the recovery of finely divided platinum by this means.

[10] Private communication, June, 1920. [11] Private communication, 1920.

SELECTED METHOD FOR THE COMMERCIAL ESTIMATION OF PLATINUM IN ORES.

In the investigation of methods for the detection and estimation of platinum in vein material, the method following has been selected as satisfactory for commercial use. It contains nothing new, but has been compiled from various sources and is similar to one used by L. W. Dunham, Irvington, N. J.[12] The manipulation has been given in detail and notes added to explain the various steps in the process. Although a separation of the platinum metals is not given, the rough group separations are included to aid in determining the value of an ore.

EPITOME OF PROCESS.

The bead obtained by usual fire-assay methods may contain gold, silver, and the platinum metals and is parted with HNO_3. The silver and most of the platinum and palladium go into solution. The residue may contain gold, iridium, rhodium, and some ruthenium and osmium, as well as platinum. The gold and the rest of the platinum are dissolved from the residue of other platinum metals with dilute aqua regia and filtered off, leaving iridium, rhodium, part of the ruthenium, and a small amount of osmium, which may be ignited and weighed. The HNO_3 solution may contain silver, platinum, and palladium. The silver is removed, as the chloride and the platinum and palladium are precipitated from the solution (which has been made slightly alkaline with Na_2CO_3), by boiling with formic acid, then filtered, ignited, and weighed. To separate the palladium from the platinum the mixed metals are treated with warm dilute HNO_3, which dissolves the palladium and leaves the platinum. The palladium is found by difference, or may be precipitated with formic acid, after evaporating to dryness with HCl, taking up with water, and neutralizing with Na_2CO_3. The aqua regia solution is evaporated to dryness twice with HCl, taken up with water, and, after the gold is removed with oxalic acid, the platinum and palladium are recovered as in the preceding process. A qualitative test is applied to a solution of the platinum precipitate.

METHOD OF PROCEDURE.

The ore in all cases is ground to pass a 100-mesh screen and when substances difficult to fuse, such as chromite and zircon, are present, the sample should be made to pass a 150-mesh screen.

When the quantity of platinum is small, it is advisable to run several samples of an assay ton each, combine the lead buttons, and scorify to about 30 grams.

An assay ton of the carefully sampled ore is thoroughly mixed with a suitable flux. As iron has a greater affinity for iridium than has lead, the old iron-nail method of treating sulphide ores should not be used. (See the table on p. 15 giving typical assay charges which have been used in the estimation of platinum in different ores.)

If there is not at least 15 times as much silver as platinum in the ore, enough of a silver salt—chloride or nitrate—is added to make

[12] Private communication, 1920.

up the deficiency. The presence of excess silver is required to render the platinum soluble in HNO_3 when parting, to assist in removing the last traces of lead in cupelling and to lessen any tendency toward loss in the cupel. The addition of silver in the form of a salt makes possible a thorough mixing with the charge, and, when reduced, the numerous silver particles are distributed in a way to be most effective in collecting the platinum.

To an unknown ore about 0.05 gram of silver chloride may be added. The charge is fluxed as in the ordinary fire assay for gold and silver, except that after the fusion has become quiet the temperature should be raised somewhat higher than is the usual practice and the heating continued for about an hour. The crucible is removed from the furnace without agitating the contents and left to cool. Cooling without agitation prevents loss of iridium, ruthenium, and osmiridium, which do not alloy with lead but sink through it and are held mechanically when the button cools. If the charge is poured, these metals are apt to be partly lost.

The button is freed from slag and cupelled at a high temperature. As the high temperature causes a loss of silver, a check or proof assay should be run at the same time to be used in correcting this error.

When platinum makes up over 1.6 per cent of the bead, the latter appears frosted to the unaided eye. The microscope will detect platinum when it constitutes 0.3 per cent of the silver bead. Considerable quantities of platinum make the bead flat and irregular. The roughness caused by iridium is of finer texture than that due to platinum. Palladium gives the surface of the bead an embosssed appearance. Ruthenium in quantity turns the surface to a bluish-black color, a black scum being left on the cupel. After cupelling, the button is then parted with HNO_3, first with 1:4, then with 1:1, and finally with 2:1 acid. If gold, silver, and all the platinum metals are present in the ore, the silver, palladium, and platinum are dissolved by this treatment, leaving the gold, iridium, rhodium, and some ruthenium and osmium. Most of the osmium and part of the ruthenium are oxidized and lost during cupellation. Part of the iridium may not collect in the silver and will be lost on the cupel.

If considerable platinum is found, some will be left still undissolved. The residue is filtered off on a 5-centimeter, ashless filter paper, ignited, and saved for the recovery of any residual platinum as well as for the determination of iridium and rhodium.

Dilute HCl is then added to the filtrate slowly, with constant stirring to precipitate the silver. After this has set over night, the silver chloride is filtered off and washed with water, acidulated with HNO_3. If the precipitate is pink, it must be redissolved and again precipitated to remove occluded platinum or palladium. Although palladium may be separated from silver by one precipitation as the chloride, in acetic acid solution, the addition of the ammonia necessary to neutralize the HNO_3 present causes a bulk of salts on evaporation, which interfere with the subsequent operations.

Evaporate the filtrate just to dryness, do not bake; take up with dilute HCl and again evaporate just to dryness, then take up with dilute HCl, transfer to a 30-cubic centimeter beaker and evaporate nearly to dryness (until there is a dry spot in the center of the

beaker). When cold, take up with a very little cold water and filter on a 5-centimeter paper to remove the last traces of silver. It is important that all the silver be removed at this point, otherwise silver would come down on boiling with formic acid and vitiate the platinum-palladium result.

The filtrate is made slightly alakaline with Na_2CO_3, formic acid added, and the solution boiled in a covered beaker until all the platinum and palladium are precipitated (about 30 minutes). These finely divided metals are filtered on a small (5-centimeter or less) paper, washed with hot water, ignited, and weighed. As the platinum metals adhere to both glazed and unglazed porcelain crucibles on ignition, "impervite" crucibles may be used to advantage for the ignition of the finely divided metals. In transferring the platinum metals from the beaker to the filter, it has been found convenient to use a small wash-bottle which will deliver a very fine stream of water. Funnels that have been cut down so that the small filter papers extend beyond the rim prevent the fine metals from creeping.

The filtrate should be boiled again with formic acid to confirm a complete precipitation of the platinum metals.

If the first parting solution is yellow or orange, the presence of palladium is indicated and the platinum may be separated from the palladium and determined. Warm the metals with dilute HNO_3 (1:4) which quickly and completely dissolves the palladium, filter, wash, ignite, and weigh the pure platinum. The palladium may be found by difference, or it may be precipitated by formic acid from the solution obtained by evaporating to dryness with HCl, dissolving in water, and neutralizing with Na_2CO_3.

The residue from HNO_3 parting (which may contain gold, iridium, and rhodium, as well as some ruthenium, osmium, and undissolved platinum) is treated with 1:5 aqua regia, dissolving the gold and platinum. The residual platinum metals are filtered off on a small filter paper, ignited (using "impervite" crucibles), and weighed. The aqua regia solution, which may contain gold and platinum, is evaporated to dryness, taken up with HCl and again evaporated to dryness, then extracted a second time with HCl, evaporated nearly to dryness, taken up with cold water, and any residual matter is filtered off. The solution is then boiled with oxalic acid until the gold is all precipitated. It is well to let the solution stand overnight before filtering off the gold. The gold is filtered off and the platinum, recovered by boiling the solution which has first been neutralized with sodium carbonate with formic acid, is then filtered off, ignited, and its weight added to that of the platinum already found.

It is advisable to check results by running a known sample along with the unknown. This known sample is made by adding to a crucible charge the approximate quantity of gold, silver, and platinum found in the ore. The check is treated in every way the same as the sample to be examined, the latter being corrected if the known sample shows a discrepancy.

The resulting platinum and palladium should be dissolved separately in aqua regia, taken to dryness twice with HCl, extracted with water, and, after each solution has been acidified with HCl, tested qualitatively, KI giving a red color with platinum and a dark-brown precipitate with palladium.

TABLE 2.—*Typical assay charges used in the estimation of platinum in different ores.*

	Silicate.	Hematite.	Limestone.	Roasted sulphide.	Black sand, largely Fe_3O_4.[a]	Chromite, 40 per cent Cr_2O_3.	Sulphides.	Sulphides with barite.
Ore, assay ton	1	1	1	1	1	1	1	1
Soda, grams	30–60	30	.....	40	.....	.....	30–60	.....
Soda bicarb., grams	.....	.....	40	.....	20	60	.....	30
Borax, grams	.....	.....	20	20	.....	.....	10	20
Borax glass, grams	5	8	.....	.....	30	20	.....	.....
Litharge, grams	60–90	70	40	40	66	35	50–150	150
Silica, grams	.....	7	5	4	.....	15	0–10	.....
Fluorspar, grams	.....	.....	.....	.....	.....	.....	.....	15
Argols, grams[b]	2.5	3.5	2.5	7	4	3	.....	.....
Niter, grams[b]	.....	.....	.....	.....	.....	.....	21	8
Silver chloride, grams	.05	.05	.05	.05	.05	.05	.05	.05
Cover	(c)	(c)	(c)	(c)	(c)	(c)	(c)	(c)

[a] This charge has been used by A. A. Hanks and was kindly furnished by him. He prefers argols to any other reducing agent because in this flux the lead collects better by the use of this reagent.
[b] The quantities of niter or reducer are only approximate and must be so chosen that a 30-gram lead button is obtained.
[c] Borax glass.

OPTIONAL COLORIMETRIC METHOD FOR ESTIMATING SMALL QUANTITIES OF PLATINUM AFTER SEPARATION BY THE PRECEDING METHOD.

Although the color developed by adding different reagents to platinum solutions has been widely used for detecting the metal, little has been published concerning the quantitative, colorimetric analysis of platinum. So far as the author has been able to ascertain the only reference to such a method in the published matter on the subject has been supplied by Hüttner and Mylius,[13] who used the colors of pure chloride solutions of the platinum metals to determine their metallic content.

The color resulting when a solution of $SnCl_2$ is added to a slightly acid solution of chloroplatinic acid is red in concentrated solutions, changing to yellow on dilution. The intensity of the color may be used to determine the quantity of platinum in a solution if certain conditions are satisfied.

The following procedure has been worked out and is to be recommended for the estimation of platinum that has been separated from the other platinum metals when the quantity of metal present is less than 0.2 milligram:

SOLUTIONS REQUIRED.

A standard HCl solution is prepared by mixing 33 cubic centimeters of HCl (concentrated) with enough distilled water to make 1 liter. The intensity of color resulting from the reduction of chloroplatinic acid by means of $SnCl_2$ is inversely proportional to the acidity (except that when the acidity is less than one-tenth normal a precipitation takes place); hence it is highly important that the acidity be the same in all samples involved. As it is convenient to have all solutions of the same acid concentration, enough standard HCl solution is best prepared to provide for this.

[13] Huttner, C., and Mylius, F. [The colorimetric determination of metals]: Ztschr. Elektrochem., Bd. 21, 1915, p. 286; Chem. Abs., vol. 9, 1915, p. 3148.

A $SnCl_2$ solution is made by dissolving 5 grams of $SnCl_2.2H_2O$ in 10 cubic centimeters of the standard HCl solution. The $SnCl_2$ solution should be freshly prepared, for it oxidizes slowly on standing.

A standard platinum solution containing 0.1 milligram of platinum per cubic centimeter is obtained by dissolving a weighed quantity of pure platinum in aqua regia, taking to dryness twice with HCl to remove the HNO_3, and then taking up with enough water (acidified with HCl) to obtain the required concentration.

The platinum solution is standardized by precipitating the platinum from a known volume (100 cubic centimeters is satisfactory) by boiling the solution, which has been neutralized with sodium carbonate, with formic acid and weighing the metal. Any slight error in results will be rendered negligible when small samples are taken for colorimetric standards.

METHOD OF PROCEDURE.

The finely divided, ignited platinum obtained by the foregoing gravimetric method is dissolved in aqua regia, evaporated just to dryness twice with HCl, and then extracted with standard HCl solution, 2 cubic centimeters being used for every 0.2 milligram or less of platinum.

A volume of 2 cubic centimeters for all quantities of platinum up to 0.2 milligram was selected because the color produced when $SnCl_2$ is added to this volume is neither too intense with 0.2 milligram to be conveniently compared nor is it so faint with 0.005 milligram as to make a determination difficult.

Quantities of the standard platinum solution, containing nearly the same amount of platinum as the unknown, are evaporated just to dryness and taken up with standard HCl, as in the previous case, 2 cubic centimeters being used for every 0.2 milligram of platinum. Two drops of the $SnCl_2$ solution are then added to each determination. A small excess of $SnCl_2$ does no harm, but enough to bring about the reduction is necessary.

The intensity of the color develops within 10 or 15 minutes and remains unchanged for several hours. After setting for 15 minutes, the solutions are transferred to test tubes of $\frac{1}{2}$ inch diameter, and the unknown is compared colorimetrically with the standard solutions.

DISCUSSION.

When less than 0.2 milligram of platinum was involved, results were obtained by the above procedure that checked more closely than those obtained by gravimetric methods. With a greater amount than 0.2 milligram of platinum, however, the gravimetric method was more accurate.

TABLE 3.—*Limit of accuracy of the colorimetric method for small quantities of platinum.*

No.	Standard solution and Pt (milligram).	Standard HCl solution (cubic centimeters).	Standard $SnCl_2$ solution (drops).	Known sample, calculated milligram of Pt.[a]	Greatest probable error.	
					Pt milligram.	Per cent.
1	0.00	2	2			
2	.005	2	2	0.005	0.00	0.00
3	.01	2	2	± .01		
4	.02	2	2	± .02		
5	.03	2	2	± .03		
6	.04	2	2	± .04		
7	.05	2	2	0.045–.055	.005	10
8	.06	2	2			
9	.07	2	2			
10	.08	2	2			
11	.09	2	2			
12	.10	2	2	.09 – .11	.01	10
13	.11	2	2			
14	.12	2	2			
15	.13	2	2			
16	.14	2	2			
17	.15	2	2			
18	.16	2	2			
19	.17	2	2			
20	.18	2	2			
21	.19	2	2			
22	.20	2	2	.18 – .22	.02	10
23	.21	2	2			
24	.22	2	2			

[a] The known sample contained the same quantity of platinum as did the standard solution in the same horizontal line.

The difference in intensity between standards whose platinum content differed by 0.01 milligram was considerable for low concentrations, but was slight when there was 0.2 milligram or more of platinum present.

In order to determine whether larger quantities of platinum could be estimated accurately by this colorimetric method, a solution containing 1 milligram of platinum was made up to 100 cubic centimeters and samples taken from it for analysis. From these results, obtained colorimetrically, the total quantity of platinum in the solution was determined, as shown in the following table:

Determination of platinum by colorimetric method.

No.	Pt in aliquot (milligram).	Pt determined colorimetrically (milligram).	Total Pt in solution, calculated (milligram).	Error, milligram of Pt.	Error, per cent.
1[a]	0.01	0.01	1.00	0.0	0
2	.05	0.045–.055	0.90–1.10	.10	10
3	.10	.09 – .11	.90–1.10	.10	10
4	.20	.18 – .22	.90–1.10	.10	10

[a] Although no difficulty was experienced in comparing No. 1 with its proper standard, the result could easily have deviated sufficiently to cause an error of 10 per cent if standards had been used whose concentration differences were small enough.

These results indicate that, although dilute solutions can be more accurately matched than concentrated, nothing is gained by diluting a strong solution and analyzing an aliquot part. The smaller error due to better comparison in dilute solutions is augmented when the total platinum content is calculated.

OTHER METHODS FOR THE DETERMINATION OF PLATINUM IN ORES.

The following methods have been used for the assay of ores:

A method often used, but not to be recommended, is known as the "difference" method. The bead obtained by the regular fire assay is parted with H_2SO_4, which dissolves the silver and leaves gold and platinum, the latter metals being annealed and weighed together. Silver is then added by inquartation and the bead parted with HNO_3, dissolving the silver and the platinum. The residual gold is annealed and weighed and the platinum found by difference. This procedure is to be avoided for the following reasons:

1. It makes no attempt to group the platinum metals in a way to assist the evaluation of an ore.

2. Parting with H_2SO_4 is unsatisfactory. Not only does boiling concentrated H_2SO_4 attack platinum, as shown by McCay[14] and by Steinmann,[15] but the procedure is also disagreeable, as hot H_2SO_4 tends to spatter. Although slightly dilute H_2SO_4 at a definite temperature may be used, the action of the several platinum metals in this treatment is uncertain.

Methods that determine platinum by "difference" have a twofold chance for error, that due to parting with H_2SO_4 and that due to the fact that the platinum may not be entirely dissolved by one treatment of the gold-platinum bead (inquartating silver and parting with HNO_3).

METHOD USED BY DART.

Dart[16] fuses the ore with a suitable flux, cupels, treats the bead with 12 per cent HNO_3, then with HNO_3 (concentrated) dissolving the silver, palladium, and platinum, the residue being weighed as gold. The silver is precipitated with HCl and filtered off. Platinum and palladium are precipitated from the filtrate after it is made slightly ammoniacal by boiling for 30 minutes with an excess of formic acid. The metals are dissolved in aqua regia, taken to dryness with HCl, and extracted with water, the platinum being precipitated by saturating the solution with NH_4Cl and adding 10 cubic centimeters of alcohol. After the solution has stood for 24 hours, the ammonium chloroplatinate is filtered off, washed with a solution of NH_4Cl, ignited, and weighed as platinum. Although this method has several good points, it is well to note that concentrated HNO_3 attacks gold[17] and that the complete recovery of very small amounts of platinum by precipitation with NH_4Cl or KCl is impossible.

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[14] McCay, LeR. W., The action of boiling H_2SO_4 on platinum: Eighth Int. Cong. App. Chem., vol. 1, 1912, p. 351.

[15] Steinmann, A., Assay of platinum: Eng. and Min. Jour., vol. 92, 1911, p. 1030.

[16] Dart, A. S., Assay of ores containing the platinum group of metals: Chem. and Met. Eng., vol. 10, 1912, p. 219.

[17] Lodge, R. W., Notes on assaying: 1906, p. 162.

METHOD USED BY CRAMPTON.

Crampton[18] gives a method used at the Boss mine for a rapid routine analysis of ore containing platinum and palladium. He estimates the extreme error to be 0.06 ounce per ton. This method weighs the platinum and palladium together. A sample of from 0.5 to 1 assay ton is mixed with a suitable flux and silver equal to 15 times the combined weight of platinum and palladium is added. The material is fused at a high temperature, cupelled at moderate heat, and parted, first with dilute and then with concentrated HNO_3. The silver is precipitated with a saturated solution of NaCl, filtered and washed, then dissolved, reprecipitated, and filtered without washing. The filtrates are made slightly alkaline with NH_4OH and boiled, after which formic acid is added in excess and the solution boiled half an hour. The platinum and palladium are filtered off without washing, dried, wrapped in lead foil, cupelled at high temperature, and weighed. This is a rapid method for the determination of platinum and palladium together, and has proved satisfactory for routine work where a high degree of accuracy is not required.

METHOD USED BY STEINMANN.

Steinmann[19] gets the lead-button by a regular fire assay, adds copper equal to the amount of platinum and gold in the sample, and cupels at a high temperature. He parts three times with a solution containing 100 parts of concentrated H_2SO_4 and 22 parts of water, heating at a temperature not to exceed 240° C. The residual metal is washed, heated, and weighed. He claims that results obtained by parting with boiling concentrated H_2SO_4 are not correct, but approximate, owing to the fact that some platinum goes into solution, while some silver and lead may stay with the platinum.

Although the above method of parting with H_2SO_4 may effect a separation of silver from gold and platinum, the action of the other platinum metals in this treatment is not definitely known.

METHOD USED BY TRENKNER.

Trenkner[20] treats the bead, which contains 10 times as much silver as platinum, with 25 cubic centimeters of H_2SO_4 (1.84 specific gravity) heated below boiling for half an hour. The residue is washed, dissolved in aqua regia, taken to dryness with HCl, extracted, and the silver filtered off. The gold is then precipitated with 1 gram of hydrazine hydrochloride in the presence of 15 cubic centimeters HCl (1.19 specific gravity).

After standing for an hour, the gold is filtered off, the filtrate evaporated to remove most of the HCl, and the platinum precipitated by boiling the solution with the addition of NH_4OH.

As given in the discussions of the "difference" method and that used by Dart, parting with H_2SO_4 is to be avoided.

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[18] Crampton, F. A., Platinum assaying at Boss mine: Min. and Sci. Press., vol. 111, 1915, p. 231.

[19] Steinmann, A., Assay of platinum: Eng. and Min. Jour., vol. 92, 1911, p. 1030.

[20] Trenkner, C., [Quantitative determination of gold, silver, and platinum]: Metallurgie, t. 9, 1912, p. 103; Chem. Abs., vol. 6, 1912, p. 1115.

METHOD USED BY SMOOT.

In a method for the determination of platinum, palladium, and gold in ores, Smoot[21] scorifies the lead-buttons from two or more one-half assay ton fusions, with six times as much silver as the combined weight of platinum, palladium, and gold, and cupels. When parted with HNO_3, the palladium goes into solution along with some platinum. The residue is dissolved in aqua regia and added to the filtrate from the palladium-silver solution, after removing the silver as follows: The silver is precipitated with HCl, the silver chloride being inclosed in lead foil and cupelled and again parted with HNO_3, the precipitation, cupelling, and parting being repeated until all the palladium is removed from the silver-chloride precipitate. The combined filtrates are evaporated to dryness with HCl two or three times, and then taken up with 5 drops of HCl and 40 cubic centimeters of water. The gold is precipitated by boiling with 3 grams of oxalic acid, left to settle over night, filtered, scorified, and determined. Five cubic centimeters of HCl is added to the filtrate and the platinum and palladium precipitated in the hot solution, which has been diluted to 150 cubic centimeters by means of H_2S. The sulphides are dissolved in aqua regia, evaporated with HCl to remove HNO_3, and the platinum recovered as ammonium chloroplatinate by the addition of NH_4Cl.

This method might be used for ores rich in the platinum metals, but its use on low-grade ores is very questionable, as it relies on the precipitation of the platinum by means of H_2S and later recovers it as ammonium chloroplatinate—a very difficult and uncertain procedure when small amounts of platinum are involved.

METHOD USED BY HANKS AND PIERS.

Hanks[22] and Piers[23] proceed as follows: A number of fire assays are run, using at least 1-assay-ton samples, with a suitable flux, effort being made to get a 30-gram lead button, enough chemically pure silver foil being added to give a proportion of 15 parts of silver to 1 part of possible platinum. After cupelling, the bead is parted with HNO_3 (1:1), and an excess of HCl added. The silver is dissolved in the HNO_3, then precipitated by HCl, and the aqua regia formed dissolves the gold, platinum, and palladium, leaving osmiridium. The silver is filtered off and, if necessary, reprecipitated to recover occluded platinum or palladium. After evaporation to dryness with HCl to remove HNO_3, oxalic acid is added to the weakly-acid solution of gold, platinum, and palladium, and, after this has boiled and settled for several hours, the gold is filtered off, the platinum and palladium being precipitated from the filtrate by specially purified zinc or magnesium. The palladium is then dissolved in HNO_3 (1:5), leaving pure platinum.

This method is similar to that given on page 19, and with proper precautions should be satisfactory. It is important that the metal used for reduction be entirely free from insoluble matter.

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[21] Smoot, A. M., Determination of platinum, palladium, and gold: Eng. and Min. Jour., vol. 99, 1915, p. 700.
[22] Hanks, Abbott A., San Francisco, Cal.: (Private communication, March, 1920).
[23] Piers, W. L., Denver, Colo.

METHOD USED BY SCHOELLER AND POWELL.

Schoeller and Powell[24] scorify the lead buttons obtained by the crucible assay of several samples into one, and then cupel with at least 10 times as much silver as platinum. Enough gold and silver are then added to give a ratio of Ag–Au–Pt, 33:10:1, and, after flattening, the bead is parted with H_2SO_4 (9:1), the residue being weighed as gold plus platinum, then wrapped in sheet lead with three times its weight of silver and cupelled. The bead is parted with HNO_3 first with that of specific gravity 1.1 and then with stronger acid 1.3. The platinum and silver are dissolved, leaving the gold, which is annealed and weighed, and the platinum is found by difference. If the gold is not pure yellow, the inquartation and the parting must be repeated. This method is similar to the "difference" method previously given and is open to the same criticism.

The following scheme by Schoeller and Powell[25] gives a method that has been used for the separation of the platinum metals, but is too involved for the commercial assay of ores:

[24] Schoeller, W. R., and Powell, A. R., The analysis of minerals and ores of the rarer elements: 1919, p. 227.
[25] Work cited.

TABLE 4.—*Separation of platinum by Schoeller and Powell method.*

Part the bead with HNO_3, first of sp. gr. 1.4, then with acid 1.3, decant, wash.

R (1): Ext. aqua regia (dil.), destroy HNO_3 with HCl, dilute, filter.		F (1): Ppt. with small excess HCl, let stand in dark till settled, filter, wash.	
R (2): Add to residue from F (6).	F (2): Boil with $C_2H_2O_4$, filter, wash.	P (4): Dissolve in min. NH_4OH, add litmus, acidify slightly with CH_3COOH, let stand in dark till settled, filter, and wash.	F (4): Evaporate dry: see F (5).
F (3): Add to residue from F (6).	P (3): Ignite, weigh gold.		
F (5): Add to residue from F (4), evap. dry, take up with water and few drops of HCl, boil with HCOOH and CH_3COONH_4 to remove Pb and Cu, filter, reject filtrate (after boiling to test complete ppt.), dissolve residue in aqua regia, evap. excess acid, dil., ppt. with $Hg(CN)_2$, filter next day.			P (5): AgCl.
P (6): Ignite, weigh Pd.	F (6): Evap. dry, ignite, add R (2) and F (3), again evap. and ignite, mix residue with dry NaCl, ignite in Cl current. Extract with alcohol filter. If not completely attacked, repeat ignition in Cl.		
R (7): Dissolve in water, ppt. with Zn or HCOOH, ignite, weigh Rh.	F (7): Evap. dry, ignite, extract with HCl (dil.), ignite in H, weigh, dissolve in aqua regia (1 : 5), filter off any residue, ignite, and weigh. Pt is obtained by difference and is confirmed by evap. filtrate to dryness, extracting with water, and adding NH_4Cl.		

METHOD USED BY DEWEY.

Dewey [26] dissolves the platinum and silver from the bead, obtained by the regular fire assay, with HNO_3 inquartating more silver and repeating the parting as long as any platinum remains undissolved. The solution is then evaporated to remove excess acid, diluted considerably, and a quantity of a dilute solution of H_2S (1 cubic centimeter of concentrated H_2S, water diluted to 15–20 cubic centimeters) added, which precipitates part of the silver and all of the platinum. The H_2S solution is added slowly with constant stirring so that, although the solution darkens, no precipitate settles out for about two hours. The precipitate is allowed to settle over night and then filtered off, dried, and ignited in a porcelain crucible. The sponge is wrapped in a small piece of lead foil and cupelled. The bead is parted by boiling with concentrated H_2SO_4, washed, dried, annealed, and weighed, the final metal being examined qualitatively.

This method has been used to separate small quantities of platinum from large amounts of silver. The difficulty of precipitating all the platinum as the sulphide and the necessity of parting with H_2SO_4 are unfortunate features of the process.

METHOD USED BY GAZE.

Gaze [27] precipitates platinum as the sulphide in the presence of $HgCl_2$ (from 5–10 per cent of the salt being added, in a solution warmed to 80–90° C.) by saturating with H_2S. The solution is heated again to aid settling and to remove excess H_2S. The sulphides are filtered and ignited to obtain the metallic platinum.

Ivanov [28] coagulates the platinum sulphide by the addition of 5 grams of $MgCl_2$ to the acid solution of the platinum salt, the H_2S causing a complete precipitation of the platinum under these conditions.

The sulphide precipitation as given in the two last methods can be considered only as a step that might be used in developing some method for the analysis of platinum ores.

SELECTED BIBLIOGRAPHY ON THE ASSAY OF PLATINUM ORES.

The following list of references has been taken from Bulletin 694 of the United States Geological Survey: Bibliography of the metals of the platinum group, by Jas. Lewis Howe and H. C. Holtz. This bulletin should be consulted if an exhaustive treatise be desired.

1816. Chaudet, Mémoire sur quelques expériences tendant à déterminer par la coupellation . . . le titre exact d'un lingot contenant de l'or du platine, de l'argent et du cuivre: Ann. chim. phys., t. 2, 1816, p. 264; Karsten, Archiv f. Bergbau, Jahrg. 11, 1826, p. 66; Ann. des mines, t. 2, 1817, p. 105.

1827. Hindl, F. X., Ueber die Probe von platinhaltenden Gold- und Silberlegirungen: Journ. prakt. Chem., Bd. 10, 1837, p. 167.

[26] Dewey, Frederic P., The direct determination of small amounts of platinum in ores and bullion: Am. Inst. Min. Eng. Bull. 64, April, 1912; Jour. Ind. and Eng. Chem., vol. 4, 1912, p. 257.

[27] Gaze, R., Bemerkungen zur quantitativen Bestimmung des Platins durch Abscheidung als Sulfid: Apoth. Ztg., Berlin, Bd. 27, 1912, p. 959; Analyst, vol. 38, 1913.

[28] Ivanov, V. N., [New method of precipitating platinum, sulphide, etc.]: Jour. Russ. Phys. Chem. Soc., vol. 48, 1916, p. 527; Chem. Abs., vol. 11, 1917, p. 766.

1878. Von Jüptner, H., Neue Methode der quantitativen Untersuchung von Gold- und Silberlegirungen: Anzeiger Akad. Wien, Bd. 15, 1878, p. 161; Bull. Soc. chim. France, t. 33, partie 2, 1880, p. 448; Ztschr. anal. Chem., Jahrg. 18, 1879, p. 104.

1879. Perry, N. W., Improved method for making platinum-alloy assays: Chem. News, vol. 39, 1879, p. 89; Eng. and Min. Jour., vol. 27, 1879, p. 29; Berg- u. hüttenm. Zeitung, Bd. 38, 1879, p. 372; Dingl. poly. Jour., vol. 240, 1881, p. 217; Ztschr. anal. Chem., Jahrg. 19, 1889, p. 83; Jour. Chem. Soc., vol. 36, 1879, p. 555; Jahresb. Chemie, 1880, p. 1196; Chem. tech. Mitth. (Elsner), Bd. 28, 1878–79, p. 35.

1880. Van Riemsdijk, A. D., Le phénomène de l'éclair dans les essais d'or et l'influence exercée sur ce phénomène par les métaux du groupe du platine: Archiv. néerland., vol. 15, 1880, p. 185; Ann. chim. phys., t. 20, partie 5, 1880, p. 66; Chem. News, vol. 41, 1880, pp. 126, 266; Ber. 13, 1880, p. 936; Berg- und hüttenm. Zeitung, Bd. 39, 1880, pp. 247, 275.

1881. Balling, C. A. M., Beitrag zur Volumetrie einiger Metalle [Influence of platinum in quartation of gold by cadmium]: Oest. Ztschr., Berg- Hüt., Bd. 29, 1881, p. 51; Chem. Ztg., Jahrg. 5, 1881, p. 113; Jahresb. Chemie, 1881, p. 1156.

1882. Van Riemsdijk, A. D. [Cupellation of gold in presence of platinum metals]: Mededeel. Labor. Rijks. Munt., 1882, No. 5; Rec. trav. chim. des Pays Bas, t. 1, 1882, p. 188; Ber. 16, 1883, p. 387.

1885. Van Riemsdijk, A. D., Sur le procédé de d'Arcet pour le dosage du platine dans son alliage avec l'argent ou avec l'or et l'argent. Sur l'essais par voie humide de l'argent tenant platine: Mededeel. Lab. Rijks. Munt., 1885, No. 6; Rec. trav. chim. Pays Bas, t. 4, 1885, p. 263; Chem. Zentralb., 1885, p. 952; Jahresb. Chemie, 1885, p. 1942; Chem. Ztg., Bd. 9, 1885, p. 1854.

1890. Matthey, E., The liquation of gold and platinum alloys: Philos. Trans., London, vol. 183 A, 1892, p. 629; Proc. Roy. Soc. London, vol. 47, 1890, p. 180; Ber. 23, 1890, R. 361; Bull. Soc. Chim., France, t. 4, partie 3, 1890, p. 824; Chem. Zentralb., 1890, i, p. 669; Chem. News, vol. 61, 1890, p. 111; Jour. Chem. Soc., vol. 58, 1890, p. 947; Jour. Soc. Chem. Ind., vol. 9, 1890, p. 624.

1892. Matthey, E., On the liquation of metals of the platinum group: Philos. Trans. London, vol. 183 A, 1892, p. 629; Proc. Roy. Soc. London, vol. 51, 1892, p. 447; Ztschr. anorg. Chemie, Bd. 2, 1892, p. 474; Jour. Soc. Chem. Ind., vol. 12, 1893, p. 448.

1895. Priwoznik, E., Ueber den Einfluss einiger Platinmetalle auf die Richtigkeit der bei den Gold-Inquartations-Proben erzielbaren Resultate: Oest. Ztschr. Berg- Hüt., Bd. 43, 1895, p. 272; Ztschr. anal. Chem., Jahrg. 35, 1896, p. 73.

——— Miller, E. H., Assay of platinum: Columbia University School of Mines, quart. 17, 1895, p. 26.

1901. Leidie, E., and Quennessen, L., Sur le dosage du platine et iridium dans les minerais de platine; Bull. Soc. Chim., France, t. 25, partie 3, 1901, p. 840; Jour. Chem. Soc., vol. 80, ii, 1901, p. 695; Chem. Zentralb., 1901, i, p. 1094.

1903. Nevel, E., L'essai du platine et ses alliages avec les métaux nobles: Ann. chim. anal., t. 8, 1903, p. 161; Jour. Chem. Soc., vol. 84, ii, 1903, p. 514; Chem. Zentralb., 1903, ii, p. 149.

1904. Sharwood, W. J., On the cupellation of platinum alloy containing silver or gold and silver: Jour. Soc. Chem. Ind., vol. 23, 1904, p. 412; Jour. Chem. Soc., vol. 86, ii, 1904, p. 450; Chem. Zentralb., 1904, 2, p. 268.

1911. Steinmann, A., Kritische Studie über das Probiren von Platin: Schweiz. Wochsch., Jahrg. 49, 1911, pp. 441, 453; Jour. suisse chim. pharm., No. 32, p. 33; Analyst, vol. 36, 1911, p. 605; Eng. and Min. Jour., vol. 92, 1911, p. 1030; idem, vol. 93, 1912, p. 228; Jour. Inst. Metals, vol. 7, 1912, p. 295; Jour. Chem. Soc., vol. 100, 2, 1911, p. 1035; Chem. Zentralb., 1911, 2, p. 1061; Chem. Abs., vol. 6, 1912, p. 201.

1911. DART, A. S., Assay of ores containing the platinum group of metals: Met. and Chem. Eng., vol. 9, 1911, p. 75.

——— Parting platinum-gold-silver bullion: Eng. and Min. Jour., vol. 92, 1911, p. 259; Jour. Inst. Metals, vol. 6, 1911, p. 349; Chem. Abs., vol. 5, 1911, p. 3210.

——— ARSEM, W. C., Separating gold, silver, and platinum (U. S. Patent 998665, July 25, 1911): Chem. Abs., vol. 5, 1911, p. 3038.

1912. DART, A. S., Assay of ores containing the platinum group of metals: Met. and Chem. Eng., vol. 10, 1912, p. 219; Jour. Inst. Metals, vol. 8, 1912, p. 323; Chem. Abs., vol. 6, 1912, p. 1723.

——— DEWEY, F. P., The direct determination of small amounts of platinum in ores and bullion: Bull. Am. Inst. Min. Eng., 1912, p. 439; Min. Sci., vol. 65, 1912, p. 274; Min. and Sci. Press, vol. 105, 1912, p. 87; idem, vol. 109, 1914, p. 20; Jour. Ind. Eng. Chem., vol. 4, 1912, p. 257; Ztschr. angew. Chem. Jahrg. 25, 1912, p. 2325; Chem. News, vol. 106, 1912, p. 8; Analyst, vol. 37, 1912, p. 281; Jour. Inst. Metals, vol. 8, 1912, p. 323; Jour. Chem. Soc., vol. 102, 2, 1912, p. 810; Chem. Zentralb., 1912, 2, p. 384; Chem. Abs., vol. 6, 1912, p. 1580.

——— KOUKLINE, E. V., [Some notes on the analysis of platinum minerals. (Full scheme of analysis)]: Rev. metall., t. 9, 1912, p. 817; Jour. Soc. Chem. Ind., vol. 31, 1912, p. 1036; Eng. and Min. Jour., vol. 94, 1912, p. 1234; Chem. Abs., vol. 7, 1913, p. 1154.

1913. RANIER, L. St., Die Fehlerquellen der platinprobe [by assay]: Oest. Ztschr. Berg-Hüt., Bd. 61, 1913, pp. 141, 155; Ztschr. angew. Chem., Jahrg. 26, 2, 1913, p. 377; Analyst, vol. 38, 1913, p. 294; Chem. Zentralb., 1913, i, p. 1542; Chem. Abs., vol. 7, 1913, p. 2170.

——— GREENWOOD, H. D., Assay method for palladium and platinum: Eng. and Min. Jour., vol. 96, 1913, p. 1175; Oest. Ztschr. Berg-Hüt., Bd. 62, 1914, p. 578; Jour. Chem. Soc., vol. 108, 2, 1915, p. 586; Chem. Zentralb. 1915, i, p. 220; Chem. Abs., vol. 8, 1914, p. 884.

——— SMOOT, A. M., Suggestions on the platinum-palladium assay: Eng. and Min. Jour., vol. 96, 1913, p. 1175.

——— GRAY, J., and TOOMBS, C., The determination of gold in the presence of iridium and allied metals in materials such as black sand: Jour. Chem. Met. Soc. South Africa, vol. 13, 1913, p. 292; Chem. Abs., vol. 8, 1914, p. 1941.

——— CLEVENGER, G. H., and YOUNG, H. W., Estimation of gold, silver, and platinum by fire assay: Min. and Sci. Press, vol. 108, 1914, p. 614; Chem. Abs., vol. 8, 1914, p. 1941.

1914. BANNISTER, C. O., and PATCHIN, G., Detection of platinum metals in cupellation beads (paper before Inst. Mining and Metallurgy): Eng. and Min. Jour., vol. 97, 1914, p. 1007; Jour. Chem. Met. Soc. South Africa, vol. 14, 1914, p. 478; Min. and Sci. Press, vol. 108, 1914, p. 146; Chem. Abs., vol. 8, 1914, pp. 1257, 3404.

——— DEWEY, F. P., Platinum assay: Min. and Sci. Press, vol. 109, 1914, p. 20; Chem. Abs., vol. 8, 1914, p. 2859.

——— HANIG, A., [Platinum. Solution with silver in assay bead]: Oest. Ztschr. Berg-Hüt., Bd. 62, 1914, p. 203; Chem. Abs., vol. 8, 1914, p. 2989.

1915. CRAMPTON, F. A., Platinum assaying at the Boss mine: Min. and Sci. Press, vol. 111, 1915, p. 231. Chem. Abs., vol. 9, 1915, p. 3042.

——— SMOOT, A. M., Determination of platinum, palladium, and gold: Eng. and Min. Jour., vol. 99, 1915, p. 700; Chem. Abs., vol. 9, 1915, p. 1441.

1918. QUENNESSEN, L., Platinum analysis: Ind. chim. rev. prod. chim., t. 5, 1918, pp. 6–7; Chem. Abs., vol. 12, 1918, p. 1367

1919. DUPARC, L., New method of analysis of platinum ores, etc.: Helvetica chim. Acta., vol. 2, 1919, pp. 324–337; Chem. Abs., vol. 13, 1919, p. 2495.

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